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Microstructural evolution and physico-chemical behavior of compacted clayey soil submitted to an alkaline plume

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Abstract: In the French concept of deep nuclear waste repositories, the galleries should be backfilled with excavated argillite after the site has been filled. Some additives like lime could be used to improve the mechanical characteristics of the argillite. After thousands of years, the degradation of the concrete lining of the galleries will generate an alkaline solution (pH value > 12) that will diffuse through the backfill. This study presents the effect of a saturated $Ca(OH)_2$ solution circulation through lime-treated sample at 60 °C for 3, 6 and 12 months, respectively. The effect of such circulation on the lime-treated Manois argillite (MA) was assessed by petrographical examination coupled to image analysis and scanning electron microscopy (SEM) equipped with energy dispersive X-ray (EDX) analyser of soil pieces. The objective of this study is to make the link among the mineralogical transformations, the textural and mechanical changes produced in the compacted clayey soil as a consequence of the alkaline solution circulation.

Key words: argillite; lime treatment; petrographical examination; scanning electron microscopy (SEM); image analysis; microstructure

1 Introduction

If the extensive use of smectite-rich bentonite as buffer material in radioactive waste disposal is largely due to the suitable properties of smectites, the interaction of highly alkaline pore water coming from the destabilization of the concrete lining may diminish the bentonite's desirable properties (its physico-chemical buffering). This is why the interaction between smectite and highly alkaline solution has become a major consideration in assessing the performance of bentonite in radioactive waste disposal. In recent years, numerous investigations were conducted in either experimental or modelling fields to evaluate the behaviors of clayey barriers in a deep geological repository for high-level radioactive wastes. To achieve this objective, it is necessary to predict mineralogical changes and transformations that are expected to occur in the barrier during the time required for radioactive wastes to reach non-hazardous radioactivity levels (tens of thousands of years to get the natural background). The alkaline solution can react with the bentonite in the proximity of concrete, inducing dissolution and precipitation of a number of phases. Bulk dissolution experiments were used to study the dissolution behavior of smectite under alkaline conditions [1–7]. Previous work on transport-reactivity systems, involving claystones in contact with hyper-alkaline fluids, predicted that the main reaction processes were dominated by ion exchange in a short time, and by dissolution-precipitation of minerals over a long time [8]. Previous studies of the reactivity of montmorillonite in alkaline solutions were focused on the collapse of expandable smectite layers, in particular on the formation of illite or illite/smectite mixed layers. However, formation of mixed layers is an intermediate step in a series of dissolution-precipitation processes. Elements released from bentonite dissolution react to precipitate several secondary phases as crystalline zeolites and amorphous calcium silicate hydrate (CSH) gels. Recrystallization of beidellite and saponite-like clay minerals was also observed [9, 10]. The alteration of the bentonite

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components neutralizes the basic solutions, and it is clearly a function of the pH value of solution, especially when the pH value is above 11 [4, 10, 11]. If the pH value is below 11, bentonite damage is limited [12]. Such modifications as dissolution and formation of some other non-swelling crystalline phases induce changes in porosity and loss of swelling and sorption capacities. These reactions will influence the porosity evolution as well. Nakayama et al. [13] showed an increase in porosity and permeability of compacted bentonite in contact with a highly alkaline solution (NaOH, a pH value of 14) tank due to montmorillonite dissolution. In the experiments performed by Karnland et al. [14], compacted bentonite was exposed to solutions with high pH values to observe changes in bentonite mineralogy and physical properties. It was pointed out that the transport was strongly affected by chemical reactions between hydroxide ions and bentonite minerals. Interaction between bentonite and hydroxide solutions leads to a significant reduction in the swelling pressure in the bentonite. The reduction seems to be due to an instant osmotic effect and a continuous dissolution of silica minerals, leading to a mass loss, and consequently a decrease in the bentonite density. Yamaguchi et al. [15] focused their experimental efforts on predicting long-term variations in hydraulic conductivity of compacted sand-bentonite mixture. Recently, Suzuki et al. [16] mentioned that smectite in bentonite can be dissolved under hyper-alkaline conditions and promotes the damage of the low hydraulic conductivity. However, data about the relationship between changes in mineralogical composition of bentonite and variations in its porosity, hydraulic conductivity or mechanical properties are scarce. The present paper examines the effect of the alkaline plume on mineralogical evolution and microstructural behavior of compacted MA treated with 4% of lime. Indeed, in the French concept, it is planned to introduce additives (bentonite or lime) in the remoulded argillite to backfill the deep galleries to enhance their geotechnical properties. The lime treatment is currently a common technique in earthworks to improve both the implementation and the long-term behavior of the soils. The basis of lime treatment is firstly cation exchange followed by a reaction with the siliceous components of clay to cause stabilization. The first reaction leads to a rapid flocculation of the clay particles by changing its cohesive nature into a friable and granular structure associated with an improvement of its strength. Stabilization is devoted to long-term additional strength development caused by precipitation of CSH and calcium aluminate gels in response to the dissolution of clays minerals in alkaline environment. Hence, the main objective of this work is the assessment of the relationship between mineralogical transformations and microstructural changes produced in the MA as a consequence of the propagation of the alkaline plume, and the assessment of the effect of lime on the argillite microstructure will be detailed. The characterization of soil structure was conducted by petrographic investigations and numerical characterization of the porous network by image analysis [17, 18]. The main difficulty is to observe clay soils in conditions which conserve the structural characteristics of their natural and hydrated states. An accurate method consists in the impregnation of the material by water-acetone-resin exchange [19]. In the present case, this step was modified since the samples were submitted to a freeze-drying procedure. The impregnation method using a resin with added UV photo-luminescence pigment was adopted to numerically characterize the macroporosity of soils on centimetric to decimetric intact samples [20].

This approach is complementary to the evaluation of the mechanical behavior presented by Cuisinier and Masrouri [21] and aims at investigating the geo-chemical processes and their link with the mechanical processes.

2 Materials and methods

The MA represents the upper part of the Callovo-Oxfordian argillite. After homogenization and being crushed in a very fine grain powder, physico-chemical analysis indicates that the MA contains 26%–32% calcite, 22%–27% rough quartz and 41%–49% clays (fraction lower than 2 μm). Clays are mainly illite, kaolinite and interstratified illite-smectite. The specific surface determined with BET is (40.4 ± 1) m^2/g. The quicklime used in that study was made of more than 97% of pure CaO. A lime content of 4% on a dry-weight basis was selected. The argillite was first wetted and left in an airtight container to reach moisture equilibrium for 2 days. Then, MA and lime were thoroughly mixed. After 24 hours, the mixture was ready for the static compaction. The lime-treated samples were sealed and maintained for 90 days before a given circulation test. The static compaction stress is about 470 kPa, the optimum dry density is 1.50 Mg/m^3, and the optimum water content is 25.0%. The main characteristics of the MA and the lime-treated MA are listed in Table 1.

Table 1 Characteristics of studied materials.

MA		
Liquid limit (%)	Plasticity index (%)	Solid density (Mg/m^3)
51.0	11.2	2.68

MA+4% lime	
Optimum dry density (Mg/m^3)	Optimum water content (%)
1.5	25.0

The main objective is to study the influence of the alkaline fluid circulation on microstructure and physico-chemical characteristics of lime-treated compacted argillite as a function of time.

1-year experiment was performed in flow-through cell specially designed to prevent any macroscopic swelling of the tested sample upon hydration. The input solution was introduced into the bottom of the cell and circulation was maintained up to 12 months. The circulation of a cement pore fluid solution was assumed by a portlandite-saturated solution (the pH value is 12.4). It was checked that the pH value remained nearly constant throughout the experiment. After compaction of the samples, the flow-through cell was put in an oven at 60 °C. The injection pressure of 40 kPa was used and measurements of current pH values were performed at the outlet of the cell. At the end of the experiment, small specimens of the compacted soil with a volume of 1–2 cm^3 were sampled.

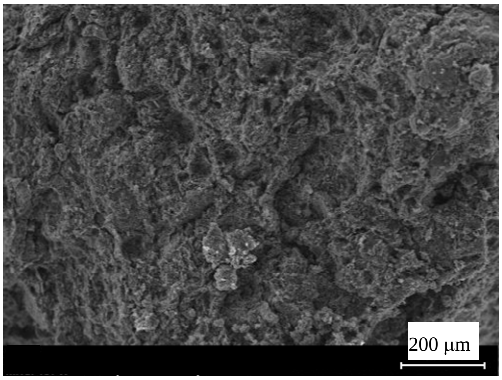
Due to the technical requirements, SEM observations must be conducted on completely dry samples. Freeze-drying was selected for our study as an alternative to oven-drying to prevent the samples microstructure from shrinking in the process of drying. Soil pieces were quickly frozen with liquid nitrogen (a temperature of –196 °C) and then placed in a freeze-drier for 72 hours for the water sublimation. SEM examinations were performed on a fresh surface obtained by dividing the dry sample. Secondary electron (SE) images allow visualisation of the morphology and the arrangement of studied materials. A gold-coating was applied for study by SEM equipped with an EDX analyser. The microscope was operated at an accelerating voltage of 15 kV and a working distance of 10 mm. In addition to SEM observations, the porous network was assessed and quantified from photographs taken under UV light of samples impregnated with a polyester resin containing a fluorescent dye (Uvitex OB, Giba-Geigy). That aims at characterizing the structural changes of the lime-treated soil by describing the impact of the water circulation experiment on the aspect of pores and fabrics of aggregates. Image analysis of soil polished sections was chosen. Images were made with a CCD camera on each section under reflected ultraviolet light, which allowed pore space to show up bright on a dark background. Each photograph was taken with the same objective (×15), and the images (10.5 mm×8.46 mm) were digitized in a rectangular grid of 1 200 pixel×1 100 pixel, with a spatial resolution of 5.88 μm per pixel. Image analysis was performed on SUN Sparc Station IPC with Visilog software. To understand the modification of the soil structure resulted from the alkaline solution leaching, these data will be compared and used as a complement to the MIP (mercury intrusion porosimetry) analysis and the mechanical behavior presented by Cuisinier and Masrouri [21].

3 Experimental results

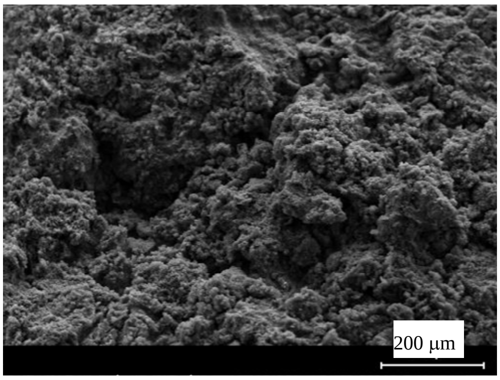
3.1 Microstructure before circulation

SEM observations of the initial compacted argillite sample showed the presence of millimetric grains of soils leaving some voids with a size reaching several hundreds of microns (Fig.1(a)). These grains are composed of aggregates with a size ranging between 100 and 200 μm. Within these grains, the internal porosity is variable and some pores can reach a radius of 50 μm or plus. At the aggregate level, we distinguished clayey minerals of smectitic type, grains of quartz, feldspars and carbonates. Figure 1(b) shows the surface state of the lime-treated MA and the intra-aggregate porosity after 7 days of treatment.

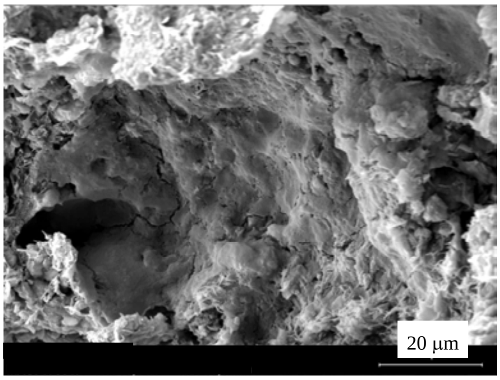
In the initial treatment phase of 90 days at 20 °C of the lime-treated MA, a strong modification was imposed on soil particles. SEM examination showed the presence of cementitious products linking the soil particles. Figure 1(c) shows the development of calcium silicates hydrates covering the soil grains and filling the inter-aggregate porosity after 90 days of treatment. These products are systematically associated with hydrated lime and traduce the in-situ reactivity of the lime with the soil minerals. In fact, these secondary compounds, corresponding to cementitious gel phase, recover part of the soil grains at the point of contact between the lime and the soil grains. That isolates the



(a) Initial state.



(b) 7 days of treatment.



(c) 90 days of treatment.

Fig.1 SE images of soil grains before circulation.

areas where the lime has no effect and the areas where the lime reacts with the soil minerals. EDX analysis of these secondary products reveals the presence of Ca and Si, and in some points these hydrates are mixed with calcium carbonates ($CaCO_3$) and portlandite ($Ca(OH)_2$). It is the same mineralogical assemblage described by Le Runigo et al. [22] for the silt treated with lime.

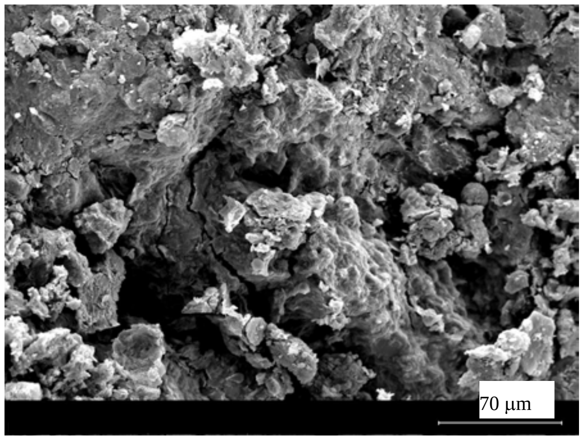
From the microstructural point of view, the lime-treatment reduces the proportion of the bigger pores (with a radius higher than 100 μm), while the cementitious products modify the surface state of the aggregates comprising the soil grains.

3.2 Influence of alkaline fluid circulation

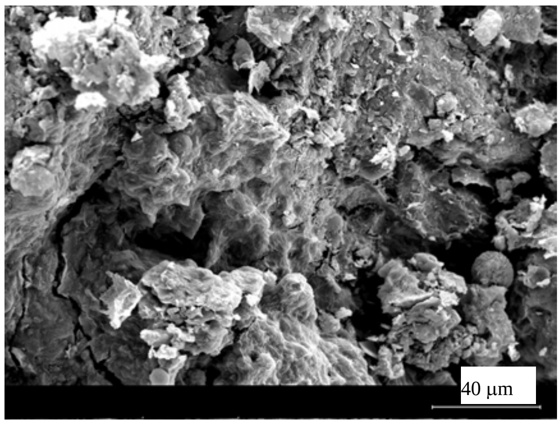
3.2.1 *SEM observations*

SEM examination of the 3-month leached specimens reveals that the secondary products formed during the treatment time are partially dissolved by the alkaline fluid circulation (Figs.2(a)–(c)). That particularly concerns the zone of fluid circulation and induces an increase in pore size. The alkaline fluid circulation induces an opening of the macroporosity resulting from the cementitious gel dissolution leaving a porous gel at the origin of the created microporosity (Fig.2(a)). The partial dissolution of the secondary products is associated with the development of some cracks within porous residual gels. In addition, the alkaline fluid circulation dissolves the soil minerals (carbonates, feldspars and clays). The mineral dissolution is only visible at the grain surface, corresponding probably to the areas of most intense circulation while the internal grains and aggregates do not show any mineral dissolution.

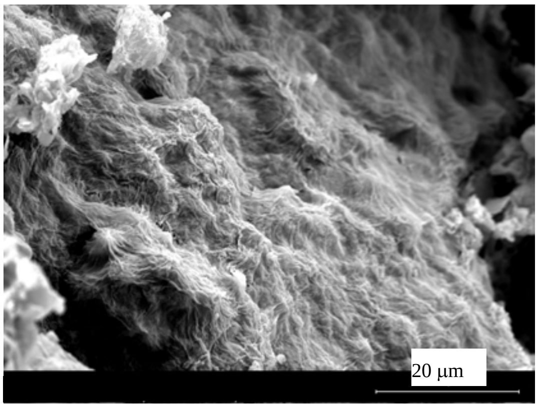
After 12 months of portlandite-saturated solution circulation, the increases have been observed since 3 months of leaching. The dissolution effect is more important and induces the formation of individual fragments of soils resulting from the dismantlement of



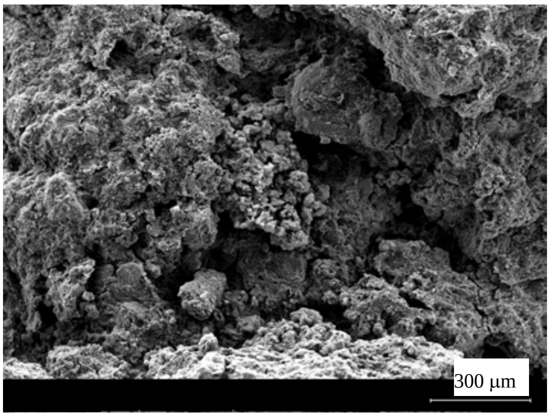
(a) During 3 months of leaching.



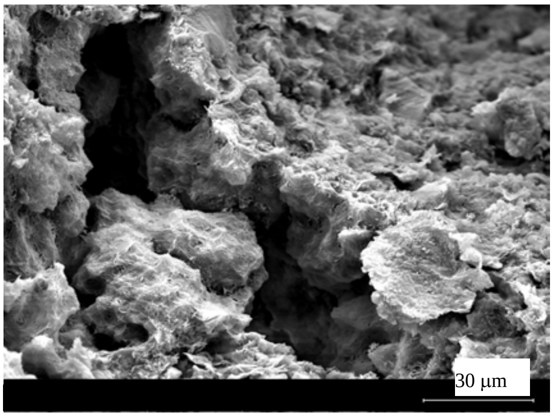
(b) Cementitious products after 3 months of leaching.



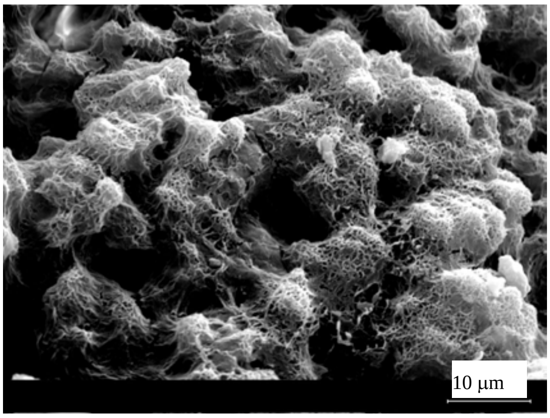
(c) Detail of cementitious compounds after 3 months of leaching.



(d) During 12 months of leaching.



(e) Detachment of soil pieces after 12 months of leaching.



(f) Dissolution of cementitious products after 12 months of leaching.

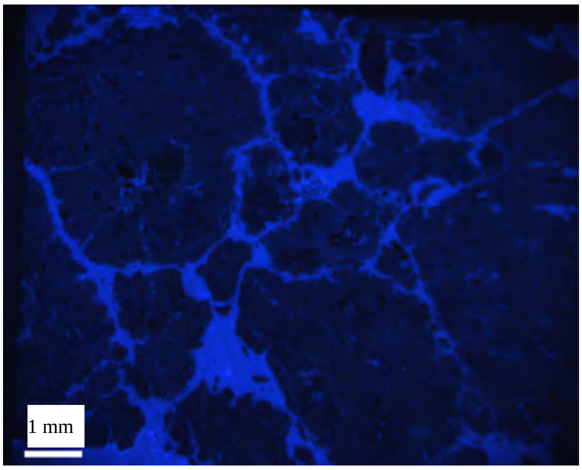
Fig.2 SE images of the lime-treated MA under alkaline fluid circulation conditions.

the soil grains and aggregates. As a consequence, the porous network grows and the mean pore size between the soil grains increases. Figure 2(f) shows that the cementitious products resulting from the lime-soil minerals interaction is highly degraded, and it also globally shows the surface state of the soil aggregates. Between 3 and 12 months of alkaline fluid circulation, the dissolution of the initial mineral compounds of the MA continues. The opening of the micropore network is evident. A number of cracks and pores are visible at the surface of the grains, traducing both the dissolution of the MA and the individualization of soil pieces or fragments as seen in Figs.2(d) and (e).

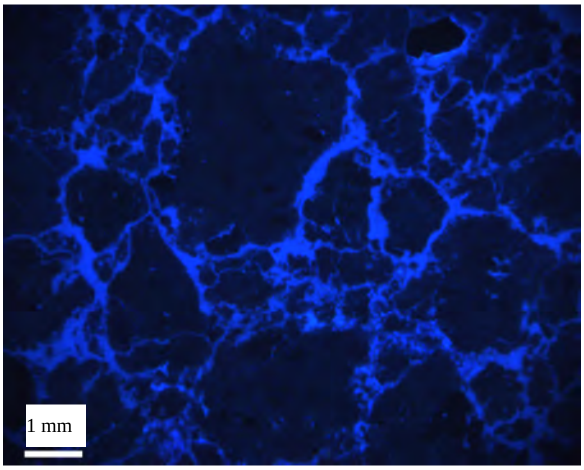
3.2.2 *Petrographical observations*

In addition to SEM observations on fresh portion of the leached soil, to understand the relationship between soil structure and water circulation effect, a detailed study of the modifications of soil structure induced by the leaching was performed. It aims at describing the impact of such alkaline circulation on the pores and fabrics of soil grains.

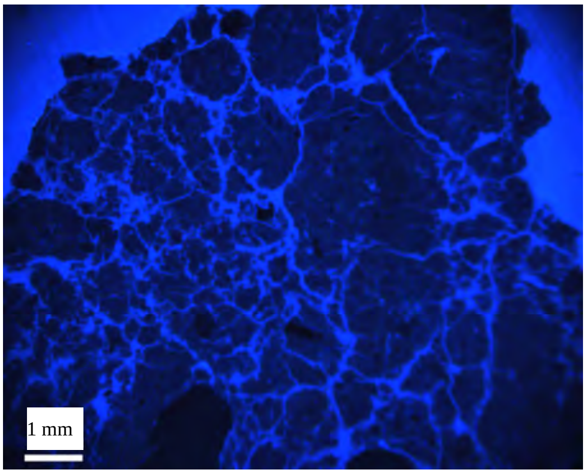
The soil impregnation reveals the 2D microstructure of the lime-treated MA. In Fig.3(a), corresponding to the initial state of the lime-treated soil, we observe a blend of soil grains, some of which are greater than 1 cm, leaving some pores around these grains. These grains are composed of aggregates separated by elongated pores while round pores are visible within the aggregates. This observation reveals the presence of big pores between the grains. The 2D microstructure of the lime-treated MA leached during 3 months by the portlandite-saturated solution at 60 °C is shown in Fig.3(b). The increase in the porosity is evident and attributed to the opening of the pores around the soil grains. The other major observation is a scatter of little pieces of soil and the development of the porosity within the grains, individualizing the aggregate composing the soil grains. The dissolution of the soil grains increases along the circulation pathways constituted by the macropores. That confirms the SEM observations, i.e. the dissolution of both the MA and the pozzolanic compounds coming from the interaction between lime and soil minerals. This increase in the opening of the porous network is clearly visible in Fig.3(c) after 12 months of alkaline fluid circulation. That particularly concerns the zone of fluid circulation and induces an increase in the pores within or between the aggregates. The mean size of the soil grains is reduced between 3 and 12 months of circulation.



(a) After compaction.



(b) 3 months of leaching.



(c) 12 months of leaching.

Fig.3 Photographs taken under UV light of the lime-treated MA.

3.2.3 *The image analysis of porosity evolution*

Global brightness reflected by blocks under UV light was used as a means to evaluate the soil porosity. Since the pore space shows up bright on a dark background, we can easily separate the voids and the solid phases. This method allows a 2D estimation of soil porosity before and after leaching. In the approach used, we distinguish two classes of pores. Based on the initial characteristics of the lime-treated MA microstructure as a blend of grains composed of aggregates, we choose to make the distinction between the inter-grain porosity corresponding to the porous network between the soil grains and the intra-grain porosity resulting from the pores within the grains, i.e. elongated pores plus isolated pores. Since the resolution is equal to 5.86 μm per pixel, we only consider in this approach that the pore larger than 5.86 μm is the macroporosity. Figure 4 presents the evolution of the MA porosity before and after leaching for 3, 6 and 12 months, respectively. The data described as the initial soil sample represent the soil porosity immediately after the compaction of the MA with the lime. These data are compared with the porosity evolution during the MA leaching.

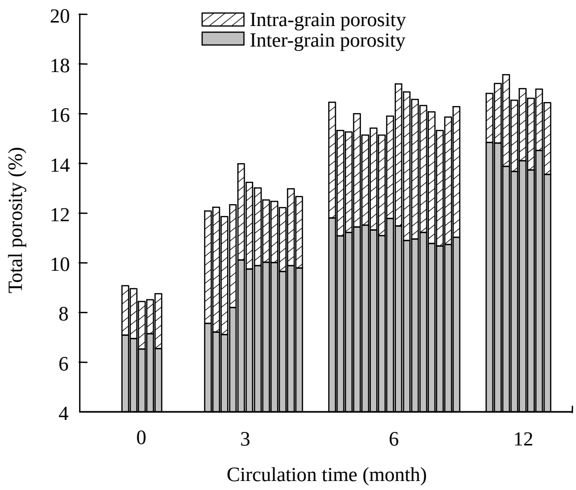


Fig.4 Evolution of the porosity of the lime-treated MA during 1 year of alkaline fluid circulation (portlandite-saturated solution at 60 °C).

The leaching leads to a progressive increase in both the intra-grain and the inter-grain porosities during 1-year experiment. During the first 3 months, we essentially observe an increase in the inter-grain porosity. Between 3 and 6 months, the increase in the intra-grain porosity is more important than that of the inter-grain porosity. The later dismantlement of the grains as a response of 1-year water circulation leads to an increase in the inter-grain porosity, and to the detriment of the intra-grain porosity.

4 Discussions

4.1 The microstructural evolution of the lime-treated MA

This works aims at assessing the effect of an alkaline fluid circulation on the MA microstructure by combining SEM and petrographical examinations coupled to image analysis.

The lime leads to a modification of the surface state of the soil grains by the secondary precipitation of amorphous hydrates. The development and the precipitation of these hydrates promote the collapse among the grains and modify the microstructure of the MA. The first level of organization in the lime-treated soil corresponds to the formation of aggregates that assemble to form some grains. That leads to the individualization of some grains of great size (Fig.3(a)), and also leads to the presence of residual voids or pores of several hundreds of microns. The porous network is essentially composed of pores around the soil grains and small pores within the grains. That corresponds to the recognition of some inter-aggregate and intra-aggregate pores.

The circulation of the portlandite-saturated solution at 60 °C during 1 year goes through preferential pathways represented by macropores, decreasing in this way the interaction with the remaining argillite buffer phases. This fluid circulation leads to a preferential dissolution of both the MA and the cementitious phases resulting from the lime treatment. As a consequence, after 3 months of leaching, the porosity increases along preferential pathways as it is clearly visible by 2D observations. From a quantitative point of view, the increase in the inter-grain porosity and the effect on the inter-grain porosity are limited.

SEM observations confirm that the reactivity of argillite minerals and the dissolution of quartz, feldspars and smectites are promoted by the alkaline fluid circulation. Montmorillonite dissolution, the mineral that contributes mainly to the reactive surface, allows the passages of different chemical species into solution. However, the precipitation of the secondary phases except the formation of portlandite ($Ca(OH)_2$) and calcium carbonates ($CaCO_3$) is not evident even if that should be checked by complementary analysis of the mineralogical content by XRD.

Between 3 and 6 months, it appears that the alkaline fluid circulation promotes the increase in the intra-aggregate porosity (Fig.4) as a consequence of a progressive dissolution of the argillite from the preferential pathways of macropores towards the internal parts of the grains. After 12 months of circulation, that leads to an important dismantlement of the grains of the small piece of soil to the detriment of the soil grains initially described in the soil. This significant evolution of the microstructure is clearly visible in Fig.12 and the distinction between inter-grain and intra-grain pores becomes less. Experimental and modelling studies of the stability of clays and clay minerals have shown that a high pH value will lead to dramatic modifications of the materials porosity (e.g. Fernandez et al. [9]).

4.2 Relationship between physico-chemical and microstructural characteristics and mechanical behavior

Cuisinier and Masrouri [21] showed by combination of MIP measurement and shear strength measurements that the alkaline fluid circulation did not produce a significant modification of the shear strength behavior of the lime-treated MA. From MIP measurement within the range of considered pore sizes (from 0.002 to 100 μm), the alkaline circulation induces a reduction in the mean micropore radius and the creation of some macropores. Again, from MIP measurement, there is no major modification at the macropore level between 3 and 12 months of alkaline fluid circulation. The major modification of the shear strength behavior of lime-treated MA occurs during the first 3 months of the fluid circulation. Longer circulation period slightly lowers the peak shear strength of lime-treated MA.

It seems very interesting to make the comparison between the microstructural evolution determined by measurement microscopic approach combined with MIP and mechanical behavior of the lime-treated MA. Petrographical observation coupled with porosity measurement from image analysis shows an increase in the total porosity of the lime-treated specimens after the alkaline fluid circulation. After 3 months of circulation, the major modification concerns the inter-grain pores of the lime-treated MA then the intra-grain ones. This approach is very complementary to the MIP measurement since the class of pores considered by image analysis concerns all the pores larger than 5.86 μm. It also shows that the treatment of the MA with 4% of lime induces the persistence of pores with mean size radius greater than 100 μm. The alkaline fluid circulation goes through these macropores, a major part of which are not considered by MIP. The recorded modifications are probably under-estimated if we only consider the MIP measurement in the present case since the petrographic images show that the major phenomena concern the macroporosity of the system. If we compare the data corresponding to the macropores distribution from MIP measurement (Fig.5) and image analysis, we note a very good agreement between both approaches. It evolves initially from 8% to around 18% after 3 months of leaching and stays constant during the rest of the alkaline circulation.

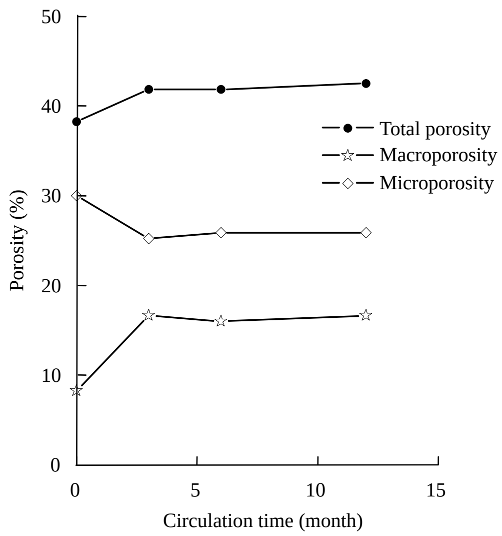


Fig.5 Evolution of the macroporosity of the lime-treated MA during 1 year of alkaline fluid circulation determined from MIP measurement.

However, even if the assessment of the microstructural evolution by petrographical observation and image analysis measurement seems to cause drastic modifications in terms of soil particles organization, shear strength is in favor of the preservation of the mechanical resistance of the material after 1 year of experimental alkaline fluid circulation at 60 °C [21].

These data should be replaced in the context of the use of the excavated argillite for the galleries backfill in deep nuclear waste repositories. 1 year of continuous leaching of portlandite-saturated solution at 60 °C modifies the microstructural organization of the lime-treated MA without modification of the mechanical behavior. It is here tested from the evolution of the shear strength.

5 Conclusions

The present study aims at assessing the effect of an alkaline fluid circulation on a lime-treated excavated argillite. This lime-treated argillite is an option for the galleries backfill of deep nuclear waste repositories. Laboratory experiments were performed to study the long-term alteration of such material with a portlandite-saturated solution at 60 °C during 1 year. SEM and petrographical examination were used to investigate the effect of such alkaline circulation at the microstructural level.

The results show that the lime-treated argillite is very sensitive to the alkaline plume as shown by the dissolution of the soil grains and the products formed during the interaction between the lime and the soil. The water circulation goes through preferential pathways of macropores existing around the soil grains, resulting from the soil preparation. The leaching continuously provokes the dissolution of the argillite and a modification of the porous network, as remarkably shown by petrographical observations. However, comparison of the data with the shear strength of the argillite shows that such modification does not alter the mechanical behavior of the argillite.

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